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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re PATENT APPLICATION of :

Knut Beekman et al. :

Serial No.: 09/548,014 : Attn: Applications Branch

Filed: April 12, 2000 : Attorney Docket No.: WLJ.051

For: A METHOD OF DEPOSITING LAYERS

CLAIM OF PRIORITY

Honorable Assistant Commissioner for Patents and Trademarks,
Washington, D.C. 20231

Sir:

Applicants, in the above-identified application, hereby claim the priority date under the International Convention of the following British application:

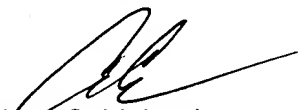
Appln. No. 9908882.5 filed April 20, 1999

as acknowledged in the Declaration of the subject application.

A certified copy of said application is being submitted herewith.

Respectfully submitted,

JONES VOLENTINE, LLP



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Dated: June 1, 2000

The Patent Office

INVESTOR IN PEOPLE

The Patent Office
Concept House
Cardiff Road
Newport
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NP10 8QQ

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Signed R. Mahoney

Dated 3rd April 2000

Patents Form 1/77

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1/77

20APR99 E440925-6 D01559
P01/7700 0.00 - 9908882.5

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

20 APR 1999

The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

1. Your reference

BKCD/DBN.101

2. Patent application number
(The Patent Office will fill in this part)

20 APR 1999

9908882.5

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Trikon Holdings Limited
Coed Rhedyn
Ringland Way
Newport
Gwent NP6 2TA

Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation

United Kingdom

7635423001

4. Title of the invention

"A Method of Depositing a Layer"

5. Name of your agent *(if you have one)*

Wynne-Jones, Laine & James

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

22 Rodney Road
Cheltenham
Gloucestershire
GL50 1JJ

Patents ADP number *(if you know it)*

1792001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

YES.

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form	
Description	8
Claim(s)	3
Abstract	0
Drawing(s)	

10. If you are also filing any of the following, state how many against each item.

Priority documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature Wynne-Jones Laine & James Date 19.4.1999

12. Name and daytime telephone number of person to contact in the United Kingdom Brian Dunlop 01242 515807

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

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b) Write your answers in capital letters using black ink or you may type them.

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e) Once you have filled in the form you must remember to sign and date it.

f) For details of the fee and ways to pay please contact the Patent Office.

"A Method of Depositing a Layer"

This invention relates to a method of depositing a layer on an exposed surface of an insulating layer of material.

It has been known for some time that the grain structure of a deposited layer can be affected by the structure of the layer on to which it is deposited. This relationship is discussed in terms of aluminium layers deposited on titanium in US Patent 5523259 and on titanium nitride layers in US Patent 5242860. Perhaps one of the most complete and recent expositions on the state of the art concerning the grain structure of metal conductors on barrier layers and how a preferential grain structure is achieved is contained in WO 99/10921. However, there is no indication in the prior art of the relationship between the structure of metallic deposited layers and insulating layers upon which they lie. Further prior art gives no indication of how such an insulating layer may be treated to improve the deposited layer structure for these purposes.

In addition to bulk interconnects another technology in which this is significant is the formation of acoustic wave devices wherein the orientation of the piezoelectric layer can be significant in the performance of the device.

Thus in a first aspect the invention consists in a method of depositing a metallic layer or layers on the exposed surface of a previously deposited insulating layer upon a substrate including treating the exposed surface with

hydrogen or a gaseous source of hydrogen in the presence of a plasma prior to the deposition of the metallic layer or layers.

Surprisingly it has been found that the exposure to hydrogen changes the structure of at least the exposed surface of the insulating layer in a sense to improve the orientation of a metallic layer and in particular a piezo electric layer subsequently deposited upon the substrate. This may be because hydrogen is implanted in the exposed surface or because the hydrogen modifies e.g. by etching the exposed surface or a combination of the two.

It is preferred that the extent of the hydrogen treatment is such that the Full Wave Half Maximum (FWHM) of the rocking curve on a preselected crystallographic plane of a deposited layer is less than 2.5°.

The plasma may be an Inductively Coupled Plasma in which case the substrate may be placed on an RF biased platen, which may be heated. Alternatively the plasma process may be Reactive Ion Etching. In the first case the process time for the hydrogen treatment may be between 35 and 25 minutes, and in the second case the treatment period may be more than 5 minutes and less than 15 minutes.

Typically the substrate will be a semiconductor such as silicon or the insulating layer will be silicon dioxide. Where the process is being used in the form of an acoustic wave device, a deposited layer will be preferably required to have a narrow x ray diffraction peak half width on (002) to function as a piezo electric thin film. This deposited

layer is preferably aluminium nitride. It is preferred that the aluminium nitride is deposited at a temperature below 500°C.

As is known in the art the FWHM rocking curve of a diffraction peak is a good indication of degree of orientation. This rocking curve is obtained by rotating a sample in an x-ray beam, which is directed at the surface being inspected. At a particular angle the curve produces a reflectance peak and by rocking the sample about that peak it is possible to determine the angle of rock needed to move the sample from half the maximum intensity on one side of the peak to the corresponding point on the other side of the peak. This angle is referred to as the FWHM measurement and the narrower the angle the better ordered the structure.

In an experiment aluminium nitride was deposited onto an underlayer of aluminium (that forms one electrode) in turn deposited upon a titanium adhesion layer upon an insulting layer of silicon dioxide. The silicon dioxide had been treated in one of three ways and the FWH rocking curve of the aluminium nitride measurement was obtained on (002).

The experimental results were as follows:

Process characteristic	Standard	Method 1	Method 2
Mode	RF biased ICP	RF biased ICP	RIE
Power Inductive coil	350W	350W	-

Substrate platen power	400W	110W	600W
Platen temperature	150°C	400°C	400°C
Process gas	30sccm Ar	50sccm H_2	300sccm H_2
Process time	32 seconds	30 minutes	10 minutes
FWHM rocking curve on (002) aluminium nitride peak	5.5	2.4	2.2

All processes here are sputter etching;

'ICP' means Inductively Coupled Plasma and includes an RF power supply predominantly inductively coupled to a plasma and an RF power supply connected to the substrate platen.

'RIE' is used here in its industry standard use; meaning that process power is predominantly or exclusively applied to the substrate holder. Any 'reactive' element to the etching is insignificant in the experimental processes reported as silicon dioxide is insignificantly reactive with hydrogen. The predominant etch method is by sputtering and due to the low mass of the hydrogen ion the etching is slow.

The standard method is a very short argon etch which is

generally used to clean the surface of a substrate prior to deposition.

The other two methods, which have been developed by the applicants, indicate the benefit of hydrogen pre-treatment, with the FWHM measurement being reduced by over half or, put another way, with an over 100% improvement in orientation.

Although, as has been mentioned above, etching may be a part of what is happening to the surface, it is noted that the etch rate of silicon dioxide using hydrogen is considerably less than argon (~21 Å/min in 'RIE' mode, 6 Å/min in ICP mode compared with 450-650 Å/min in the 'standard' process").

In the light of this, it is considered more likely that hydrogen is implanted into the surface of the substrate improving the grain structure of that surface or it may be that the hydrogen in the surface creates favourable conditions for the grain orientation of the subsequently deposited layer. This process, whilst it enables the formation of bulk SAW devices is commercially viable in

spite of its long process times. However, the applicants have also observed that an 80mm titanium film which is sputtered onto a silicon wafer in a cryogenically pumped vacuum system that has stood idle for a long period of time (say 10 hours or more) gives a Ti<002>:<011> XRD ratio that is high (>10:1). A vacuum would, in those conditions, have a high hydrogen content due to the well known inability of cryogenic pumps to pump hydrogen well.

Another aspect of the invention is therefore to treat the first or subsequent metallic layers of a multilayer structure with atomic hydrogen, typically in a plasma. This could most conveniently be done simultaneously with a metallic sputtering process e.g. titanium, titanium nitride, titanium oxide, tungsten, tungsten nitride, tantalum, tantalum nitride, aluminium, aluminium alloys, copper, aluminium nitride.

The sputtering process requires a plasma to generate the argon ions required for the process from the argon gas supplied to the vacuum process chamber. Hydrogen additions

in controlled small quantities would be ionised by the electric fields present within the vacuum vessel (or could be previously ionised): Thus atomic hydrogen would be incorporated within at least the surface of the sputtered film causing its crystallographic structure to be preferentially modified.

This structural modification of at least the surface enables a higher proportion of a preferential crystal orientation in a subsequently deposited metal conductor. Thus a layer of a metallic barrier structure lying between an insulating layer and a conducting layer could be crystallographically modified by the use of this hydrogen treatment thus causing layers further deposited upon it to have a preferential structure. In addition or alternatively the layers requiring this preferential crystallography could be sputtered themselves in the presence of hydrogen or subsequently treated with atomic hydrogen. The preferential structure is characterised by having increased functional capabilities due to its more regularly ordered

crystallography e.g. a reduced susceptibility to electromigration and is frequently characterised by having a higher proportion of <111> crystal orientation.

CLAIMS

1. A method of depositing a metallic layer on an exposed surface of previously deposited insulating layer on a substrate including treating the exposed surface with hydrogen or a gaseous source of hydrogen in the presence of a plasma, prior to or during deposition of the metallic layer.

2. A method as claimed in Claim 1 wherein the hydrogen treatment modifies the exposed surface.

3. A method as claimed in Claim 1 wherein hydrogen is implanted in the exposed surface.

4. A method as claimed in any one of the preceding claims wherein the extent of the hydrogen treatment is such that the x-ray diffraction peak half width on a crystallographic plane of a deposited metallic layer is narrowed.

5. A method as claimed in Claim 4 where the metallic layer is aluminium nitride.

6. A method as claimed in any one of the preceding claims wherein the plasma is an Inductively Coupled Plasma.

7. A method as claimed in Claim 6 wherein the substrate is placed on an RF biased platen.

8. A method as claimed in Claim 7 wherein the platen is heated.

9. A method as claimed in any one of Claims 1 to 4 wherein the plasma means is Reactive Ion Etching.

10. A method as claimed in Claim 9 wherein the treatment time is less than 15 minutes.

11. A method of depositing a metallic layer including the modification of its crystallographic structure by the use of atomic hydrogen.

12. A method as claimed in Claim 11 wherein the metallic layer is deposited by sputtering and molecular hydrogen is added to a metallic sputtering process.

13. A method as claimed in Claim 11 or Claim 12 wherein the metallic layer is titanium or a titanium alloy and the modification includes the enhancement of the <002> crystallographic orientation of the titanium or alloy.

14. A method as claimed in Claim 11 or Claim 12 wherein the

metallic layer is copper, copper alloy, aluminium, or an aluminium alloy or titanium nitride the modification includes the enhancement of the <111> crystallographic orientation of the metallic layer.

15. A method of forming an acoustic wave device including depositing a metallic layer in accordance with a method as claimed in any one of the preceding claims.